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Development of a Unified Theory for Predicting the Behaviour of Glassy Waste Products (Flyash, Fumed Silica and Slags) in Concrete and to Develop New Compositions of Concrete for Offshore Applications

for

Canada Centre for Mineral and Energy Technology
Energy, Mines and Resources
Ottawa, Ontario

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Materials Division
Glass and Ceramics Centre

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1. INTRODUCTION

The overall objectives of the EMR Flyash program are to:

i) Develop a unified theory to predict the behaviour of glassy waste products (flyash, silica fume, and slags).

ii) On the basis of the formulated theory, develop new compositions of concrete for offshore and other marine applications.

The specific objective of the first phase is to study the kinetics and mechanism pertaining to the reaction of glassy waste materials with leaching agents including water, alkalies, and other solutions.

2. PROJECT STATUS

i) The project is proceeding according to plan.

ii) The project is on budget.

iii) There are no problem areas for which assistance from the Crown is required.

3. PROGRESS-TO-DATE

3.1 Literature Assessment

Work on the chapter on Hydration Kinetics of Glassy Waste Materials was continued.

3.2 Experimental

The experimental work carried out this month has been described in detail in Appendix A. The main results of the investigations were the following:

(a) Glass content determinations were carried out on the cordierite model system and on Wabamun fly ash using the Differential Intensity Technique. The technique is promising for systems in which the

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crystalline phases in both the partially and fully crystallized samples are the same and present in the same ratios. The technique as it was developed for organic polymers appears not applicable to fly ashes without modifications.

(b) Hydration studies in NaOH solution of pH = 7-11 show that Wabamun fly ash and standard slag behave generally as in deionized water. Dalhousie fly ash lowers the pH of the leaching solutions presumably because it removes OH^- through complexation with $Fe^{3+}_{(aq)}$.

3.3 Subcontract

Work on the evaluation of the cementitious properties of non-ferrous slags is continuing according to the directions provided by Dr. E. Douglas of the Department of Energy, Mines and Resources (CANMET).

4. FUTURE WORK

Efforts during the month of September will be directed towards correlation of the data of leaching trials and with structural parameters.

Mark van Roode

M. van Roode
Research Scientist

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APPENDIX A

Experimental Work

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1. INTRODUCTION

The experimental work carried out during the month of August involved a preliminary evaluation of the Differential Intensity XRD method for glass content determinations and hydration studies on two fly ashes and one blast furnace slag using sodium hydroxide solutions.

2. EXPERIMENTAL

2.1 Glass Content Determination - Differential Intensity XRD Method

An assessment of the literature on glass content methodology was included with Monthly Report No. 67075-83-06. The differential intensity method described by Wakelin et al. for polymers (Section 2.3.2(d)) involves the calculation of the percentage crystallinity from an integral or correlation index. These indices are defined as

$$\text{Integral Index : } C_i = \frac{\sum_{2\theta_o}^{2\theta_m} (I_u - I_a)}{\sum_{2\theta_o}^{2\theta_m} (I_c - I_a)} \qquad (1)$$

$$\text{Correlation Index : } (I_u - I_a) = C_c\,(I_c - I_a) + B \qquad (2)$$

where I_u: intensity of the (partly crystalline) sample at designated 2θ-values.

I_a: intensity of the amorphous polymorph at these 2θ-values.

I_c: intensity of the crystalline polymorph at these 2θ-values.

The intensity values are measured over small 2θ-increments (at the most 0.5^o).

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A preliminary investigation was carried out to determine the feasibility of glass content determinations for fly ashes and slags using the differential intensity XRD method. As for the non-crystalline XRD technique the technique was first used to measure the glass content of systems with a known glass content. The system used was cordierite ($2MgO.2Al_2O_3.5SiO_2$).

From a rapid XRD scan of the glassy polymorph it was determined that the amorphous halo was in the 2θ range of $10.0-40.0^{o}$. Scattering intensities for cordierite glass, α-cordierite and mixtures containing predetermined glass contents are listed in Table 1 and Table 2, respectively.

Percentages crystallinity and glass contents of the two cordierite mixtures (A and B) are given in Table 4. It can be seen that the agreement between the known and the calculated values is good when the correlation index is used but rather poor when using the integral index. This observation agrees well with the findings of other workers (e.g. See L.E. Alexander, X-Ray Diffraction Methods in Polymer Science, Wiley-Interscience, N.Y., 1969, pp.177-188).

Figures 1 and 2 show graphical representations of I_u-I_a versus I_c-I_a for the mixtures containing 20 and 70% cordierite glass, respectively. Linear relationships can be plotted although the correlation coefficients, r=0.722 and 0.772, are not very strong. It can be seen that the scatter in the data is substantial for the more positive values of I_u-I_a and I_c-I_a, i.e. for the 2θ values with significant crystalline scattering. The data are more nearly linear for 2θ values where the crystalline scattering is rather weak.

Figure 3 shows XRD scans reconstituted from the counting intensities of Table 2. It can be seen that the peak positions for the 2θ values with strong crystalline scattering agree closely for the α-cordierite and the glassy-crystalline mixture.

On the basis of the results for the cordierite system the two differential intensity techniques have been applied to Wabamun fly ash for

which fully vitreous and redevitrified analogues had been prepared. Table 3 lists counting intensities while the I_u-I_a versus I_c-I_a relationship and reconstituted XRD scans are shown in Figures 4 and 5, respectively. Table 4 gives crystallinity indices and glass contents. It can be seen that the correlation index which is believed to be the more reliable measure of crystallinity is unreasonable giving a crystalline content of -1.0%. The correlation coefficient (r=-0.0508) shows that I_u-I_a and I_c-I_a are not correlated.

The reconstituted XRD scans (Figure 5) illustrates the origin of the poor correlation. The crystalline phases in the as received fly ash and its redevitrified analogue are clearly different.

The correlation index, C_c, was therefore recalculated leaving out the strongly scattering 2Θ values of the fly ash and its redevitrified analogue (counting intensities marked with an * in Table 3). The recalculated glass content (83.6%) is close to the value found using the integral index (82.0%). This value is closer to the glass content calculated with the QXRD technique (76.1%) than the value calculated using the Non-Crystalline Scattering Method (62.1%). It would be interesting to use the Differential Intensity Method in this modified form to establish glass content values for comparison with alternate techniques.

2.2 Hydration Kinetics

Hydration studies were carried out on Wabamun and Dalhousie fly ashes and Standard slag using NaOH as a leaching medium. The pH range surveyed to-date was the range pH=7 to pH=11.

Leaching was carried out on 1.00g of the "as received" sample in 100 mL of $NaOH_{(aq)}$ solution.

pH=7

The fly ashes and slags behaved as observed earlier (see Monthly Reports 67075-84-07 and 67075-84-09). Wabamun reaches pH≈10 within 30 min.

which drops after 2 hrs to reach pH≈9 after 24 hrs. Dalhousie shows an initial pH<4 which gradually increases to pH>7 after 24 hrs. Standard slag initially behaves like Wabamun but the pH drop is less from 2 to 24 hrs.

pH=8

The behaviour is similar to that observed for pH=7.

pH=9

Wabamun and Standard Slag behave as for pH7 and 8. Dalhousie has an initial pH<5 but reaches pH=7 within 10 min.

pH=10

Wabamun behaves as for pH=7-9. The pH for Standard Slag is remarkably constant while Dalhousie reaches a pH=7.5 within 10 min, which goes up to pH=8 from 40 min to 4-1/2 hrs after which a slight drop to pH=7.7 is observed.

pH=11

All three curves show very similar behaviour with the pH hovering about pH=11. Dalhousie shows a noticeable drop in pH to reach a value of pH=9.6 after 24 hrs.

These initial results in moderately alkaline NaOH solutions show that

(a) Wabamun fly ash and Standard slag show the same behaviour for pH=7-10 as in deionized water. At pH=11 it appears that the solution is little effected by these materials.

(b) Dalhousie tends to lower the pH of these alkaline solutions but the effect decreases with increasing pH (although it is still noticeable for leaching times in excess of 6 hrs at pH=11). It is likely that the lowering of the pH is the result of the reaction between $Fe^{3+}_{(aq)}$ and $Fe^{3+}_{(glass)}$ with $OH^{-}_{(aq)}$ ions in solution.

TABLE 1

Scattering Intensities of Cordierite Glass (I_a), α-Cordierite (I_c) and a Mixture of 20% Cordierite Glass and 80% of α-Cordierite (I_u)

2Θ	I_a	I_c	I_u	I_c-I_a	I_u-I_a
Standard	35483	35610	34316		
10.0	9515	20242	39669	10727	30154
10.5	9497	54058	34259	44561	24762
11.0	9525	6568	6962	-2957	-2563
11.5	9664	6219	6366	-3445	-3298
12.0	9919	6290	6585	-3629	-3334
12.5	10384	6473	6444	-3911	-3940
13.0	10675	6912	6515	-3763	-4160
13.5	10984	6645	6649	-4339	-4335
14.0	11438	7430	6999	-4008	-4439
14.5	12285	6715	6834	-5570	-5451
15.0	12891	6547	6722	-6344	-6169
15.5	13763	6906	7062	-6857	-6701
16.0	14535	10260	9074	-4275	-5461
16.5	15562	18987	10373	3425	-5189
17.0	16799	7538	7756	-9261	-9043
17.5	18118	8142	9366	-9976	-8752
18.0	19542	25688	64542	6146	45000
18.5	21306	9535	11909	-11771	-9397
19.0	23489	19405	19860	-4084	-3629
19.5	25417	11805	11398	-13612	-14019
20.0	27991	19765	13757	-8226	-14234
20.5	30705	19648	16069	-11057	-14636
21.0	33033	10908	13803	-122125	-19230
21.5	35685	46573	90730	10888	55045
22.0	38336	12514	15697	-25822	-22639
22.5	40538	9434	13373	-31104	-27165
23.0	42024	9255	13489	-32769	-28535
23.5	43526	11448	15154	-32078	-28372

TABLE 1 continued

2θ	I_a	I_c	I_u	I_c-I_a	I_u-I_a
24.0	43712	9299	13704	-34413	-30008
24.5	44015	9171	13940	-34844	-30075
25.0	43934	10895	14227	-33039	-29707
25.5	43624	39178	24008	-4446	-19616
26.0	41688	174712	77852	133024	36164
26.5	40034	34330	33853	-5704	-6181
27.0	38565	15588	16035	-22977	-22530
27.5	36616	10922	13733	-25694	-22883
28.0	34718	19057	21171	-15661	-13547
28.5	33806	61954	81137	28148	47331
29.0	31666	12689	16831	-18977	-14835
29.5	29832	97420	184773	67588	154941
30.0	28467	10072	12048	-18395	-16419
30.5	27091	19699	15226	-7392	-11865
31.0	25946	27986	18035	2040	-7911
31.5	24697	12541	11382	-12156	-13315
32.0	23314	8266	9073	-15048	-14241
32.5	22150	7919	9004	-14231	-13146
33.0	21330	21732	16317	402	-5013
33.5	20525	11795	11665	-8730	-8860
34.0	19838	23567	36978	3729	17140
34.5	19203	9708	8963	-9495	-10240
35.0	19156	22337	17601	3181	-1555
35.5	17779	15950	12146	-1829	-5633
36.0	16774	36227	18908	19453	2134
36.5	16204	16710	13660	506	-2544
37.0	16007	16192	13483	185	-2524
37.5	15783	8330	7937	-7453	-7846
38.0	16046	6992	9762	-9054	-6284
38.5	16984	18282	25567	1298	8583
39.0	14148	12842	10642	-1306	-3506
39.5	14047	12872	15931	-1175	1884
40.0	14203	14019	10856	-184	-3347
				-227,885	-155,184

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TABLE 2

Scattering Intensities of Cordierite Glass (I_a), α-Cordierite (I_c) and a Mixture of 70% Cordierite Glass and 30% of α-Cordierite (I_u)

2Θ	I_a	I_c	I_u	I_c-I_a	I_u-I_a
Standard	35483	35610	34911		
10.0	9515	20242	18983	10727	9468
10.5	9497	54058	24692	44561	15195
11.0	9525	6568	8053	-2957	-1472
11.5	9664	6219	8108	-3445	-1556
12.0	9919	6290	8094	-3629	-1825
12.5	10384	6473	8282	-3911	-2102
13.0	10675	6912	8428	-3763	-2247
13.5	10984	6645	8728	-4339	-2256
14.0	11438	7430	9121	-4008	-2317
14.5	12285	6715	9246	-5570	-3039
15.0	12891	6547	9585	-6344	-3306
15.5	13763	6906	10360	-6857	-3403
16.0	14535	10260	11411	-4275	-3124
16.5	15562	18987	12955	3425	-2607
17.0	16799	7538	12452	-9261	-4347
17.5	18118	8142	14479	-9976	-3639
18.0	19542	25688	31323	6146	11781
18.5	21306	9535	16435	-11771	-4871
19.0	23489	19405	21909	-4084	-1580
19.5	25417	11805	18885	-13612	-6532
20.0	27991	19765	21464	-8226	-6437
20.5	30705	19648	24065	-11057	-6640
21.0	33033	10908	24808	-22125	-8225
21.5	35685	46573	50890	10888	15205
22.0	38336	12514	28846	-25822	-9490
22.5	40538	9434	28716	-31104	-11822
23.0	42024	9255	30674	-32769	-11350
23.5	43526	11448	31888	-32078	-11638
24.0	43712	9299	31686	-34413	-12026

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TABLE 2 continued

2Θ	I_a	I_c	I_u	I_c-I_a	I_u-I_a
24.5	44015	9171	32139	-34844	-11876
25.0	43934	10895	31621	-33039	-12314
25.5	43624	39178	34653	-4446	-8971
26.0	41688	174712	54582	133024	12894
26.5	40034	34330	38393	-5704	-641
27.0	38565	15588	29480	-22977	-9085
27.5	36616	10922	27437	-25694	-9179
28.0	34718	19057	28644	-15661	-6074
28.5	33806	61954	44495	28148	10689
29.0	31666	12689	25152	-18977	-6514
29.5	29832	97420	76650	67588	46818
30.0	28467	10072	21341	-18395	-7126
30.5	27091	19699	21856	-7392	-5235
31.0	25946	27986	21919	2040	-4027
31.5	24697	12541	18691	-12156	-6006
32.0	23314	8266	19270	-15048	-4044
32.5	22150	7919	16655	-14231	-5495
33.0	21330	21732	19500	402	-1830
33.5	20525	11795	16559	-8730	-3966
34.0	19838	23567	23083	3729	3245
34.5	19203	9708	14795	-9495	-4408
35.0	19156	22337	18738	3181	-418
35.5	17779	15950	15387	-1829	-2392
36.0	16774	36227	17256	19453	482
36.5	16204	16710	15244	506	-960
37.0	16007	16192	14526	185	-1481
37.5	15783	8330	12363	-7453	-3420
38.0	16046	6992	13288	-9054	-2758
38.5	16984	18282	18208	1298	-1224
39.0	14148	12842	12686	-1306	-1462
39.5	14047	12872	14761	-1175	714
40.0	14203	14019	12947	-184	-1256
TOTAL				-227,885	-123,522

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TABLE 3

Scattering Intensities of Wabamun Fly Ash Glass (I_a) its Redevitrified Analogue (I_u) and Wabamun Fly Ash (I_u)

2θ	I_a	I_c	I_u	I_c-I_a	I_u-I_a	
Standard						
10.0	7904	6436	7733	-1468	-171	
10.5	7957	6750	7678	-1207	-279	
11.0	7687	5435	7629	-2252	-58	
11.5	7604	5287	7780	-2317	176	
12.0	7777	5255	8006	-2522	229	
12.5	7901	5105	7891	-2796	-10	
13.0	8239	5134	8169	-3105	-70	
13.5	8156	7004	8301	-1152	145	
14.0	8462	5109	8490	-3353	28	
14.5	8561	4662	9056	-3899	495	
15.0	9096	5018	9166	-4078	70	
15.5	9593	4763	9789	-4830	196	
16.0	10221	4670	12477	-5551	2256	
16.5	10901	4596	12010	-6305	1109	
17.0	11592	4746	13848	-6846	2256	
17.5	12853	5868	12200	-6985	-653	
18.0	13744	6900	13047	-6844	-697	
18.5	14937	6340	14303	-8597	-634	
19.0	16309	7792	15123	-8517	-1186	
19.5	17769	5870	16072	-11899	-1697	
20.0	19248	6532	17865	-12716	-1383	
20.5	20731	11340	28353	-9391	7622	*
21.0	22126	17926	20790	-4200	-1336	*
21.5	23575	125113	23061	101538	-514	*
22.0	25040	52819	21414	27779	-3626	*
22.5	25820	16306	21125	-9514	-4695	*
23.0	26732	10926	21452	-15806	-5280	
23.5	27189	23732	21744	-3457	-5445	*
24.0	26966	24272	21353	-2694	-5613	*
24.5	27141	13514	20648	-13627	-6493	

TABLE 3 continued

2Θ	I_a	I_c	I_u	I_c-I_a	I_u-I_a	
25.0	26986	7869	21139	-19117	-5847	
25.5	26063	14823	23074	-11240	-2989	
26.0	25454	11650	36178	-13804	10724	*
26.5	25247	13350	36464	-11897	11217	*
27.0	23841	9701	20643	-14140	-3198	*
27.5	22935	53516	19919	30581	-3016	*
28.0	22306	63031	19511	40725	-2795	*
28.5	21273	16461	18242	-4812	-3031	
29.0	20324	9764	17744	-10560	-2580	
29.5	19616	13309	17344	-6307	-2272	
30.0	18789	12634	16968	-6155	-1821	
30.5	18016	19887	16568	1871	-1448	
31.0	17344	14550	18191	-2794	847	*
31.5	16457	12474	15440	-3983	-1017	
32.0	16021	9321	15601	-6700	-420	
32.5	15509	7346	15983	-8163	474	
33.0	14572	13114	24677	-1458	10125	*
33.5	14086	12725	15493	-1361	1407	
34.0	13499	6536	12314	-6963	-1185	
34.5	13107	6879	11841	-6228	-1266	
35.0	12601	11141	15101	-1460	2500	
35.5	12104	40334	12784	28230	680	
36.0	11567	15204	11525	3637	-42	*
36.5	11055	10609	14855	-446	3800	
37.0	10711	9731	11340	-980	629	
37.5	10395	8310	9844	-2085	-551	
38.0	11211	7314	9565	-3897	-1646	
38.5	11149	6858	9030	-4291	-2119	
39.0	9884	6613	10925	-3271	1041	
39.5	9683	7761	12184	-1922	2501	
40.0	9823	6436	10347	-3387	524	
TOTAL				-88,988	-16,032	

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TABLE 4

Glass Contents Determined with the
Differential Intensity Method

	Cordierite		Wabamun
	A	B	
% Glass (known)	20	70	?
Integral Index (C_i)	0.681	0.542	0.180
Crystallinity (C_i)%	68.1	54.2	18.0
% Glass (C_i)	31.9	45.8	82.0
Correlation Index (C_i)	0.795	0.278	-0.0104
			(0.164)
Correlation Coefficient (r)	0.722	0.772	-0.0508
			(0.529)
Crystallinity (C_i)%	79.5	27.8	-1.0
			(16.4)
% Glass (C_c)	20.5	72.2	101.0
			(83.6)

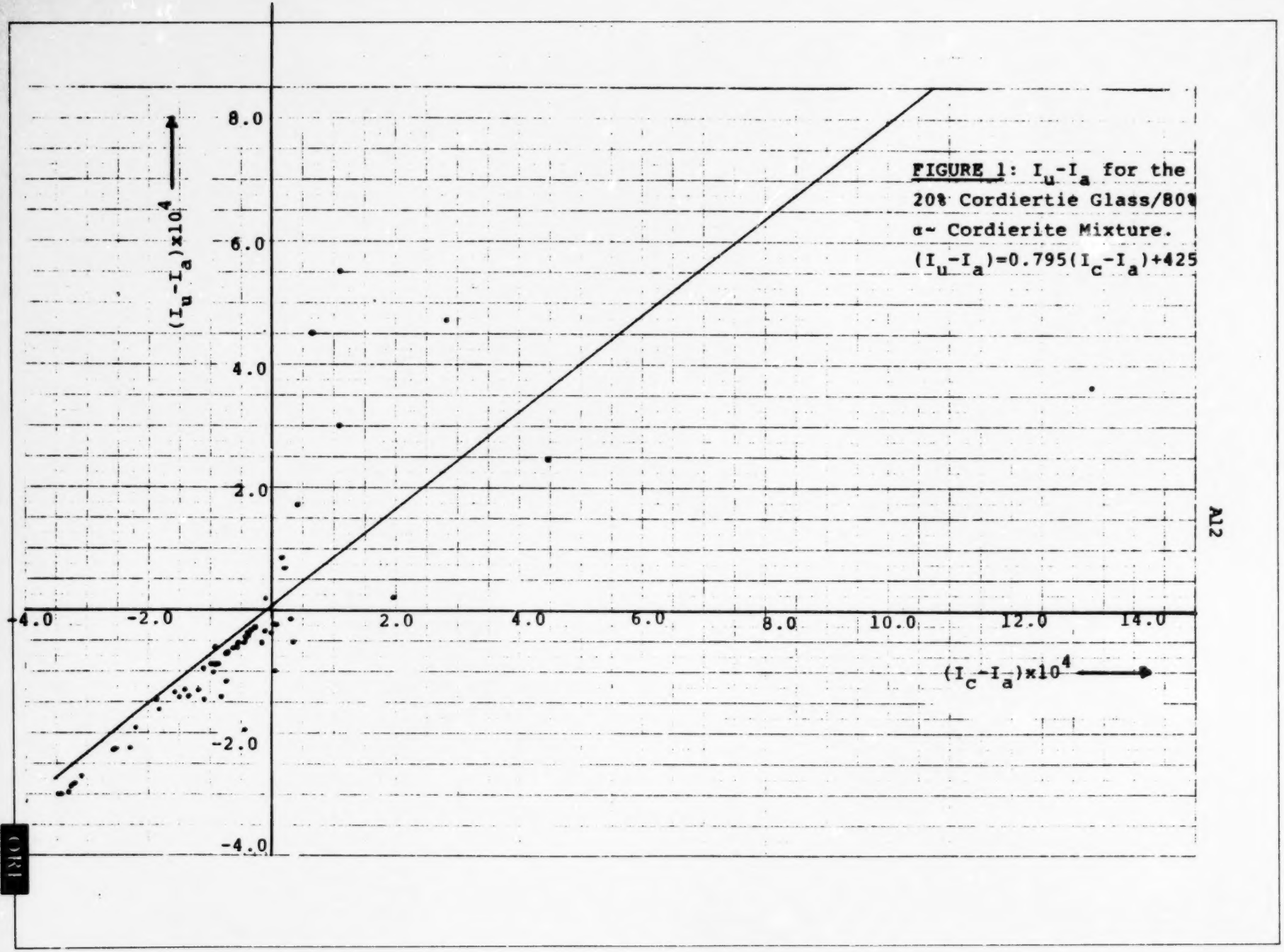

FIGURE 1: I_u-I_a for the 20% Cordiertie Glass/80% α~ Cordierite Mixture.
$(I_u-I_a)=0.795(I_c-I_a)+425$

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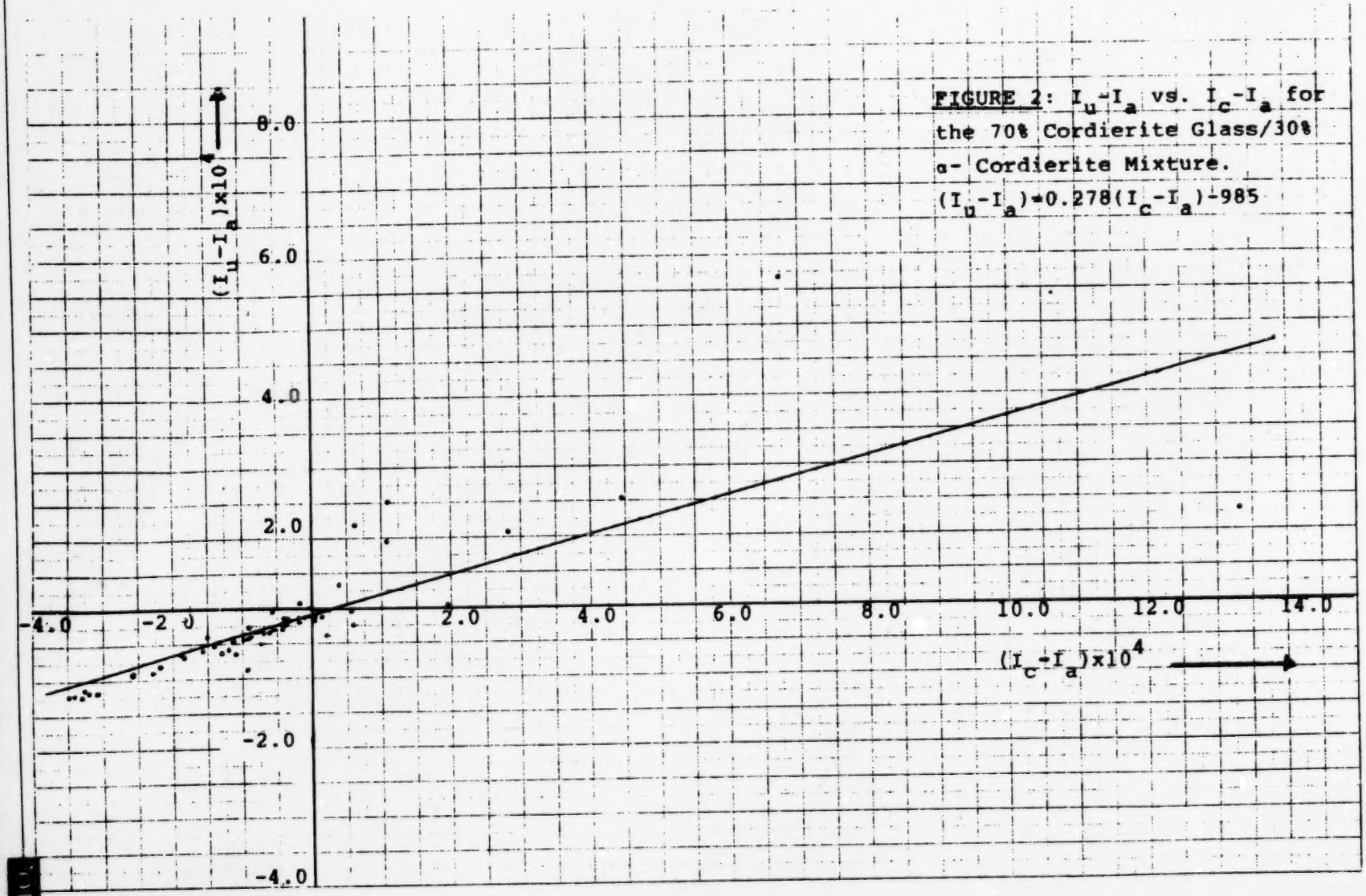

FIGURE 2: I_u-I_a vs. I_c-I_a for the 70% Cordierite Glass/30% α- Cordierite Mixture.
$(I_u-I_a)=0.278(I_c-I_a)-985$

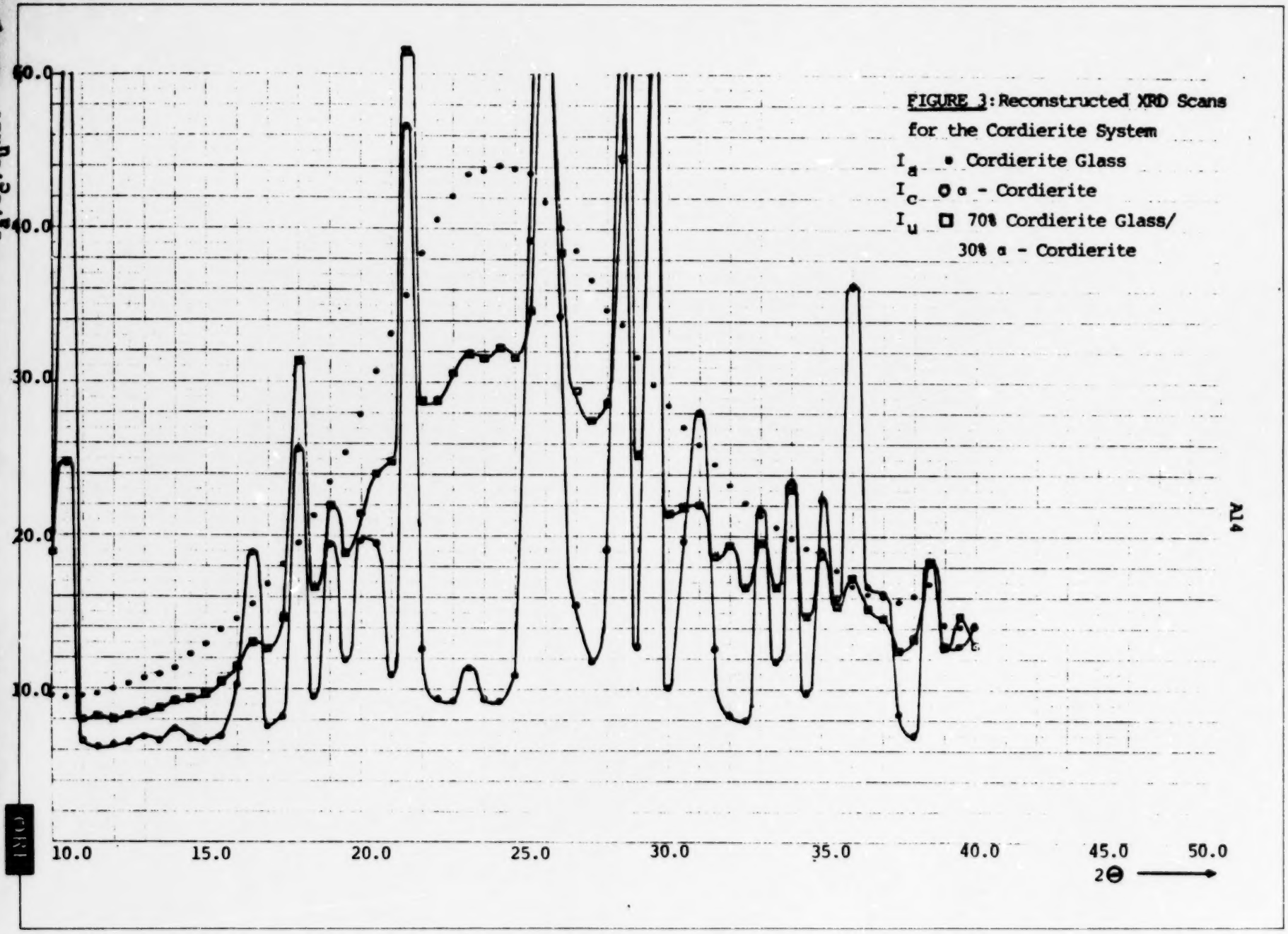
FIGURE 3: Reconstructed XRD Scans for the Cordierite System
I_a • Cordierite Glass
I_c ○ α - Cordierite
I_u □ 70% Cordierite Glass/ 30% α - Cordierite
60.0
40.0
30.0
20.0
10.0
10.0
15.0
20.0
25.0
30.0
35.0
40.0
45.0
50.0
2Θ
A14

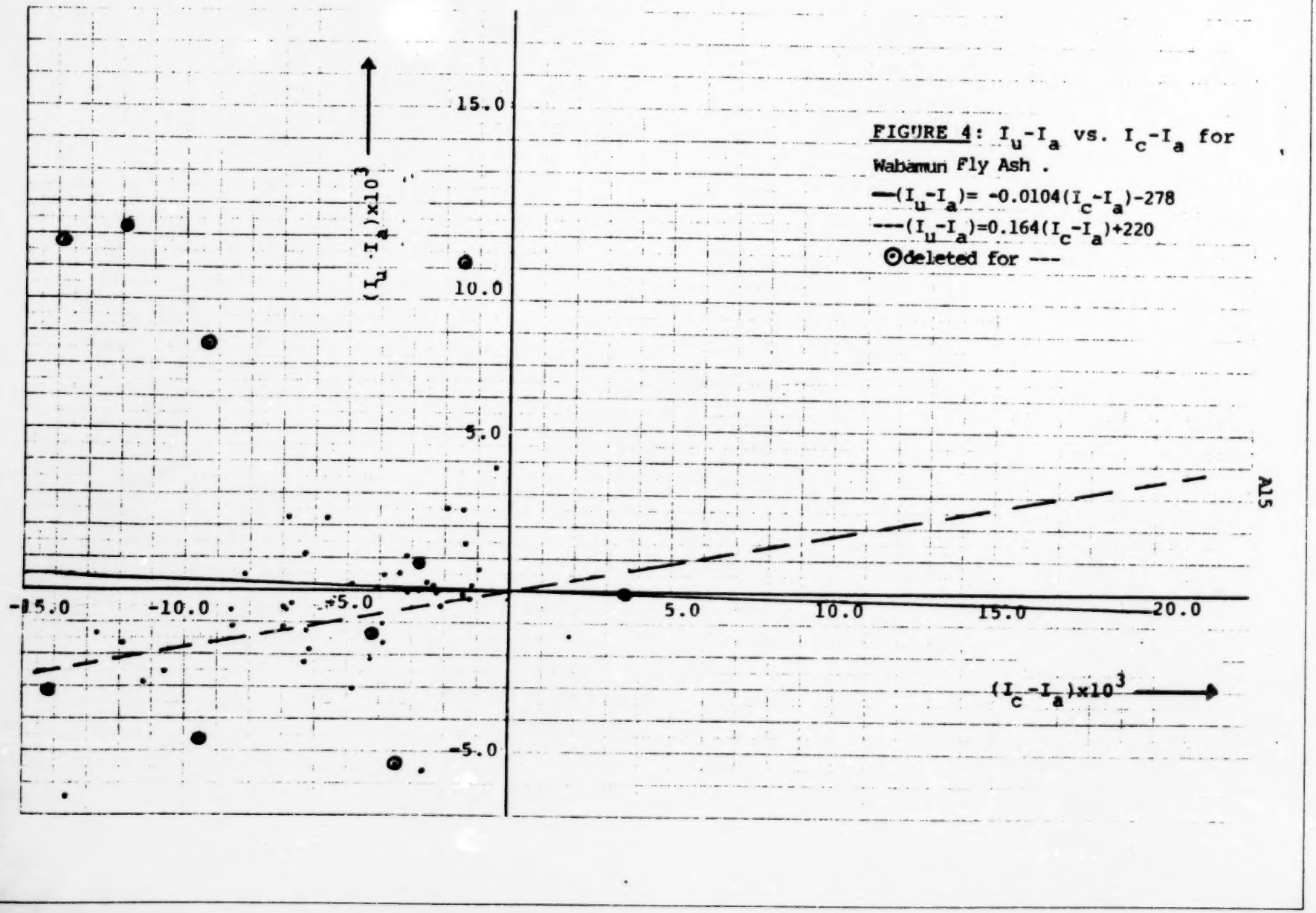

FIGURE 4: I_u-I_a vs. I_c-I_a for Wabamun Fly Ash .

—$(I_u-I_a)= -0.0104(I_c-I_a)-278$

---$(I_u-I_a)=0.164(I_c-I_a)+220$

⊙deleted for ---

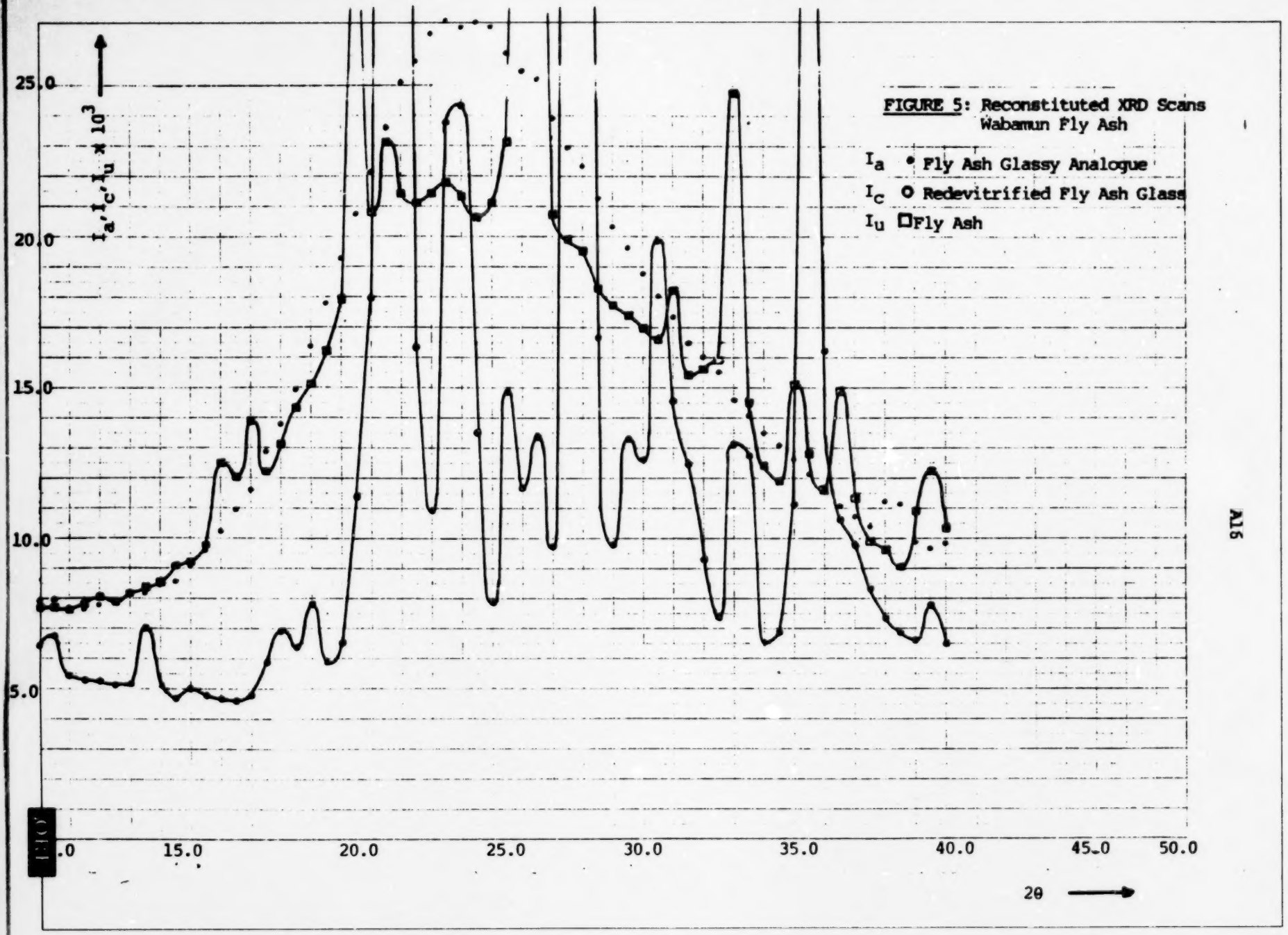

FIGURE 5: Reconstituted XRD Scans
Wabamun Fly Ash

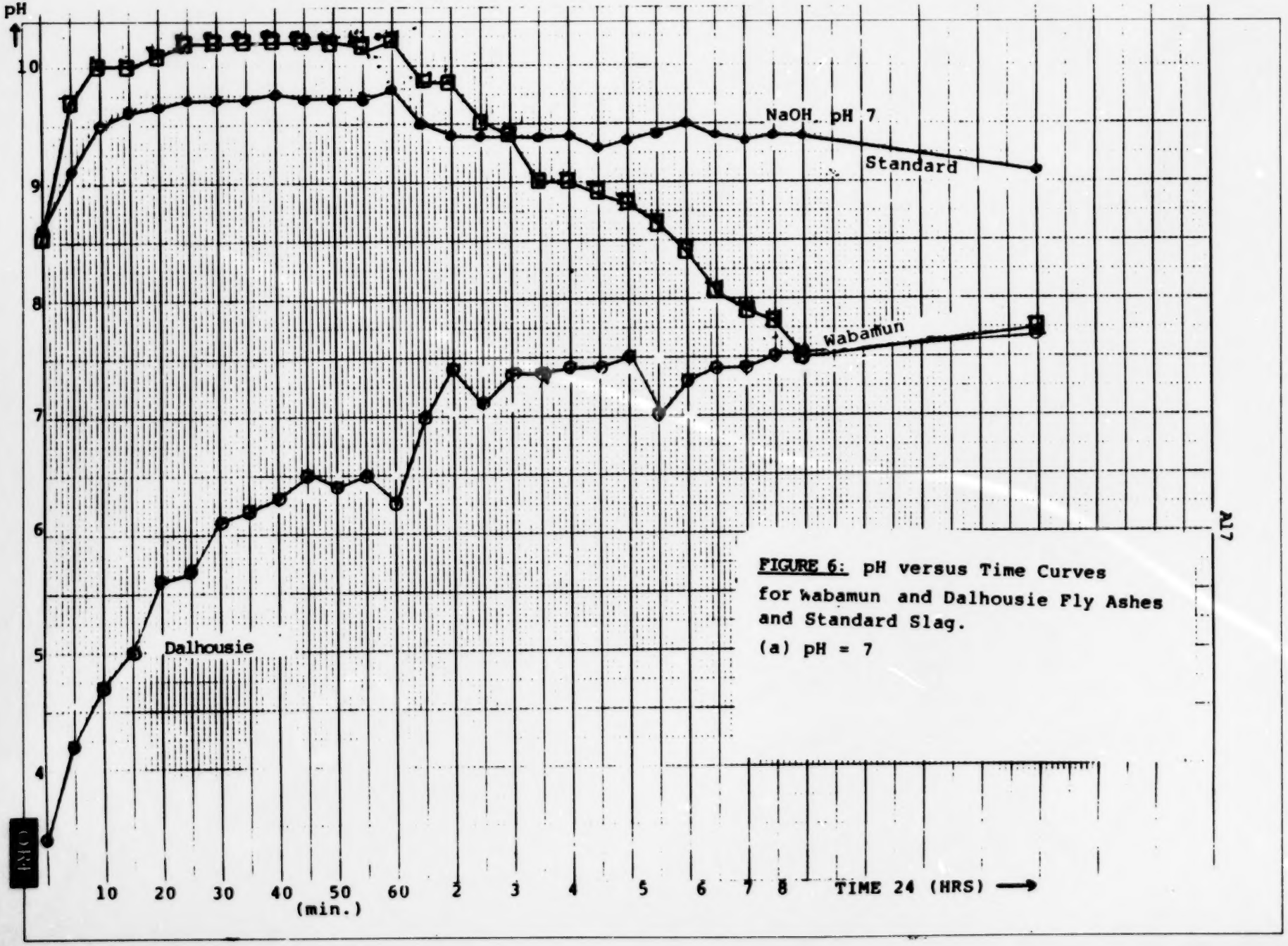

FIGURE 6: pH versus Time Curves for Wabamun and Dalhousie Fly Ashes and Standard Slag.
(a) pH = 7

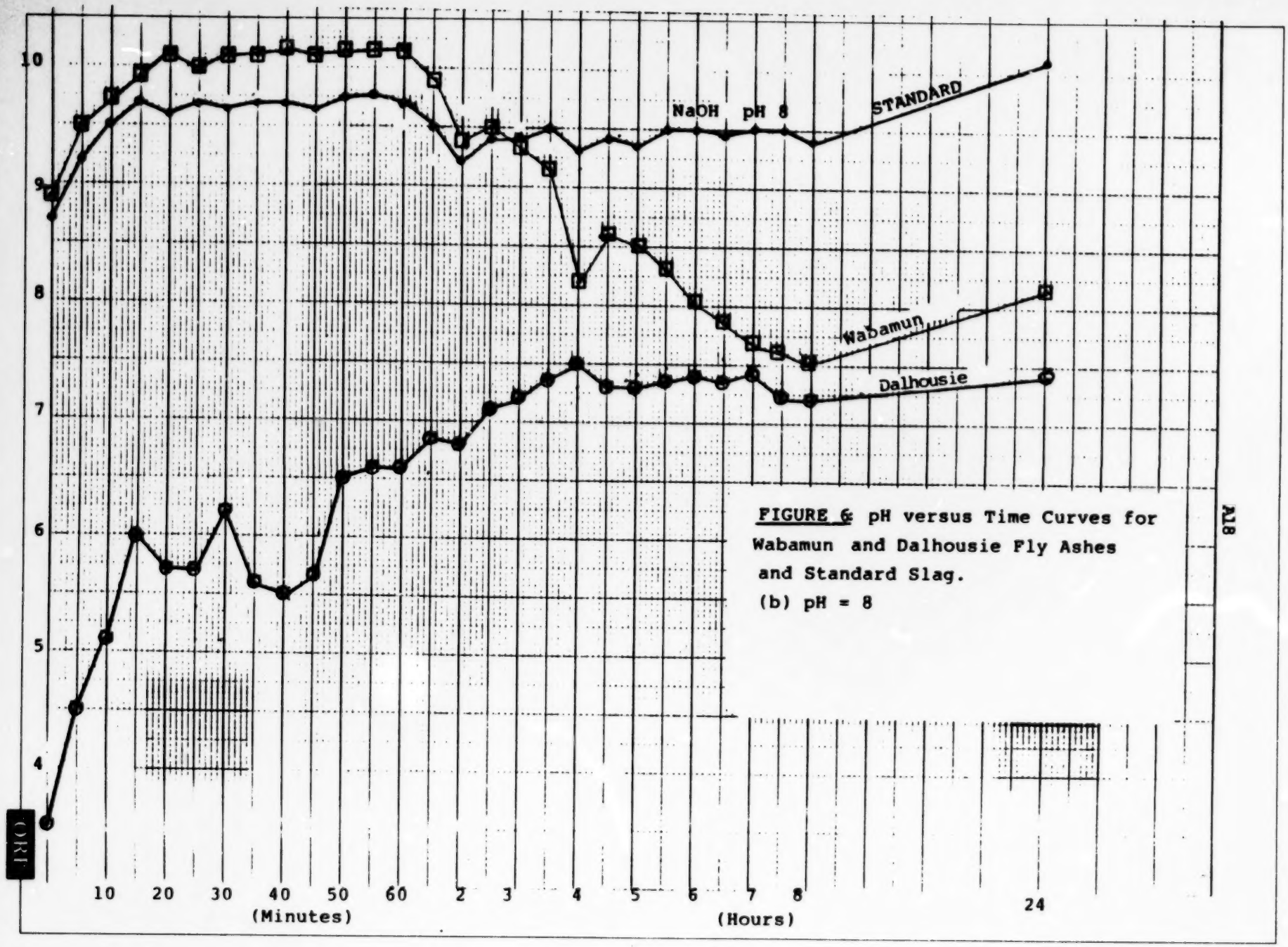

FIGURE 6: pH versus Time Curves for Wabamun and Dalhousie Fly Ashes and Standard Slag.
(b) pH = 8

A18

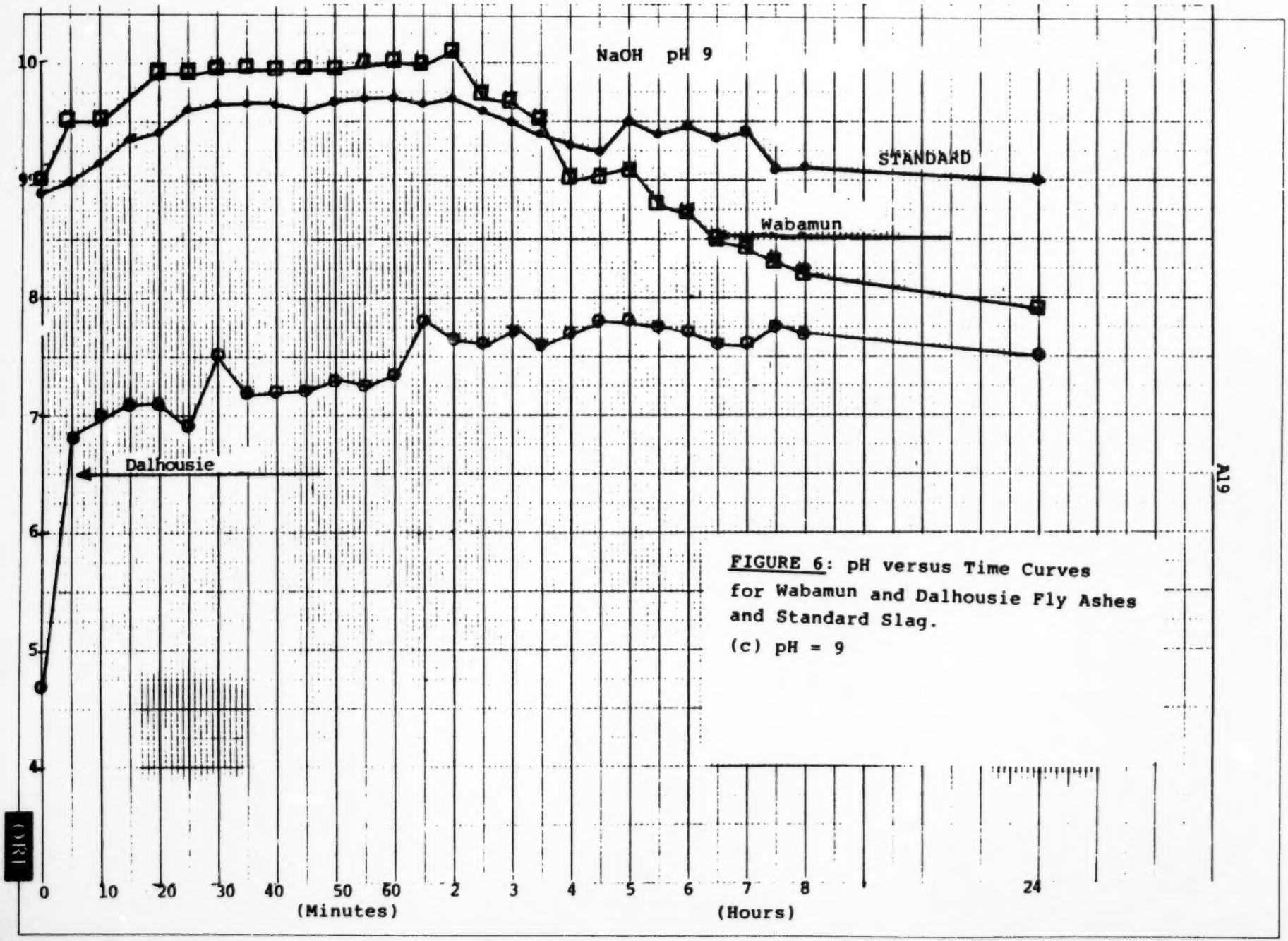

FIGURE 6: pH versus Time Curves for Wabamun and Dalhousie Fly Ashes and Standard Slag.
(c) pH = 9

CRI

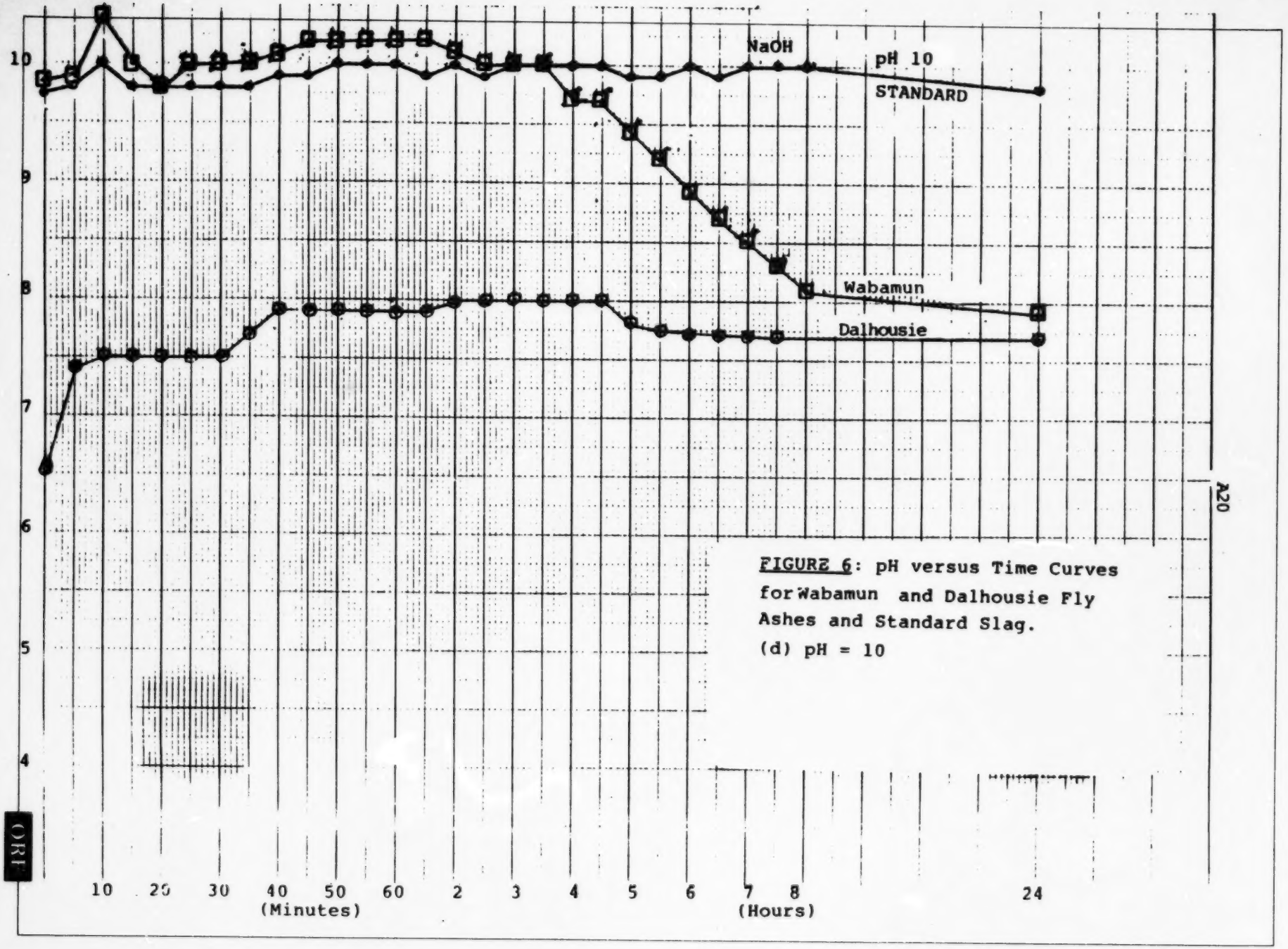

FIGURE 6: pH versus Time Curves for Wabamun and Dalhousie Fly Ashes and Standard Slag.
(d) pH = 10

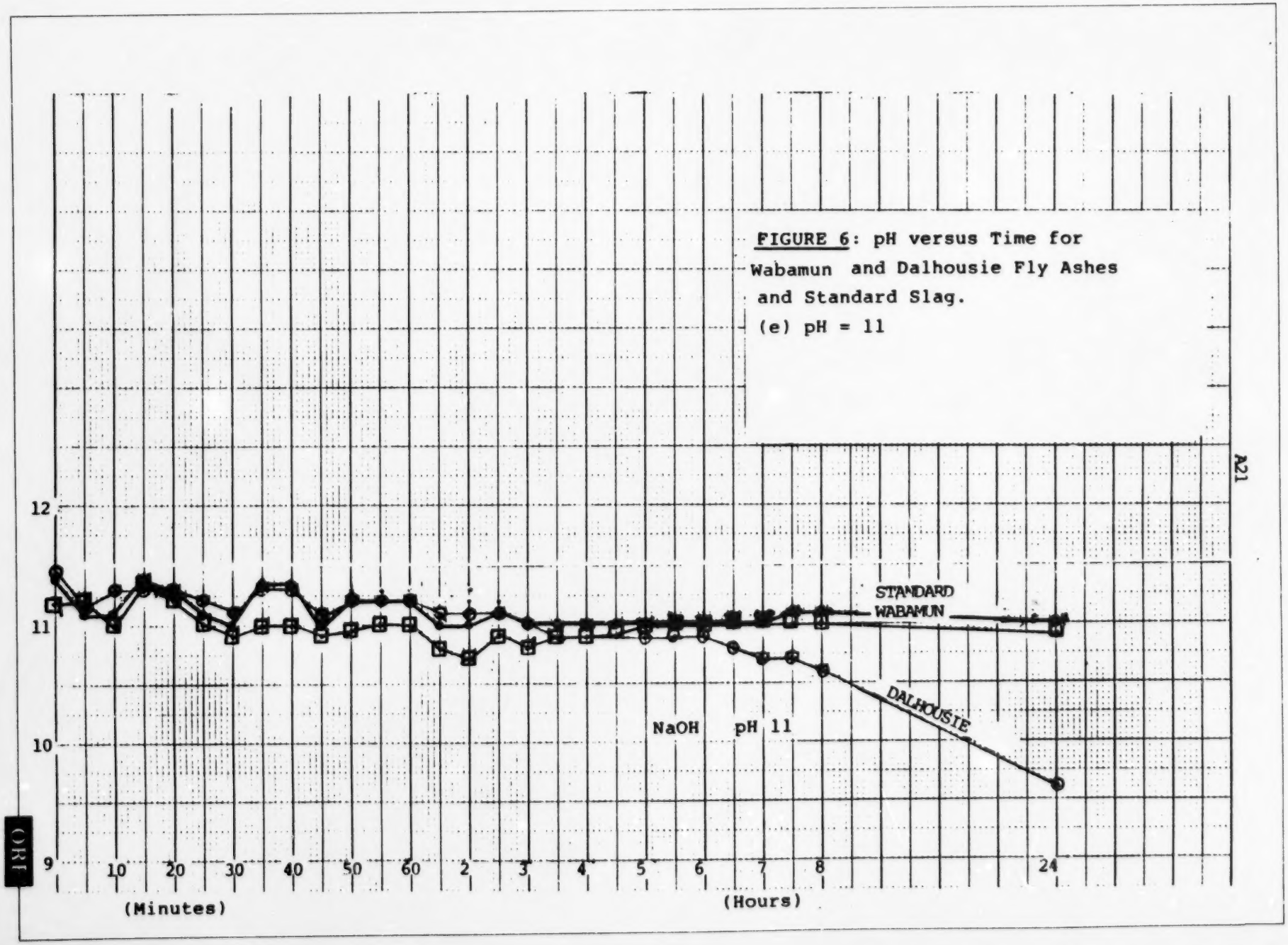

FIGURE 6: pH versus Time for Wabamun and Dalhousie Fly Ashes and Standard Slag.
(e) pH = 11

ORI

DSS File No. 15SQ.23440-3-9193

Contract Ser.No. ISQ83-00162

DATE _____ REVISION NO. _____

PLAN OF WORK AND COST (APPENDIX)

CONTRACTOR: Ontario Research Foundation

TITLE: To develop a Unified Theory to Predict the Behaviour of Flyash Fumed Silica and Slags in Concrete

CONCURRED BY (INITIALS):

CONTRACTOR: _____

NRC: _____ SSC: _____

WORK PERFORMANCE — Activities / Milestones

EXPENDITURE — JOUR

% COMPLETE	WORK BREAKDOWN (ACTIVITIES/MILESTONES) — MONTHS		1 Nov. 83	2 Dec. 83	3 Jan. 84	4 Feb. 84	5 Mar. 84	6 Apr. 84	7 May. 84	8 Jun. 84	9 Jul. 84	10 Aug. 84	11 Sept. 84	12 Oct. 84	EST COST ACTIVITY/ MILESTONE	FOR NRC USE ONLY
80	Critical Assessment of Literature	P	←				→								15,000	
		A/E	←				→									
100	Selection of Candidate Materials & Prelim. Eval.	P		←			→								10,000	
		A/E		←			→									
80	Development and Application of Methodology	P			←						→				26,000	
		A/E			←						→					
50	Hydration Kinetics & Mechanism Study	P				←								→	28,000	
		A/E				←								→		
50	Analysis & Correlation of Results	P										←		→	12,000	
		A/E										←		→		
	Final Report.	P												←	4,430	
		A/E												←		
		P														
		A/E														
		P														
		A/E														
		P														
		A/E														
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		P														
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		P														
		A/E														
		P														
		A/E														
		P														
		A/E														
	PERSON DAYS - PLANNED	P	3	7	45	40	45	26	26	26	26	26	26	25		
	- ACTUAL & EXPECTED	A/E	3	7	36	39	34	25	38	40	25	24				
	BY MONTH - PLANNED	P	1500	3300	12000	11,630	12,000	8,000	8,000	8,000	8,000	8,000	8,000	7,000	95,430	
	- ACTUAL & EXPECTED	A/E	1396	3272	12,504	9,553	19,704	6,317	9,821	10,558	6,210	7,257				
	CUMULATIVE - PLANNED	P	1500	4800	16300	28430	40430	48,430	56,430	64,430	72,430	80,430	88,430	95,430		
	- ACTUAL & EXPECTED	A/E	1396	4,668	17,172	26,725	46,430,	52,747	61,806	72,163	78,373	85,631				

A - UNDER APPLICABLE MONTHS

ORF